DETERMINATION OF CARBOXYLIC ACIDS, CARBOHYDRATES

ICP-AES IN A SINGLE CHROMATOGRAPHIC RUN

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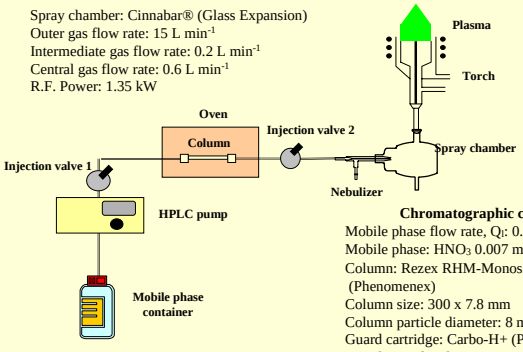
1. Introduction

The determination of carbohydrates, carboxylic acids and metals is a subject of great interest in the field of food science because their presence and concentration can influence on food quality. Besides the study of the profiles of these compounds can permit the discrimination between different varieties of a product. Unfortunately, the usually employed chromatographic detectors (*i.e.*, refractive index, fluorescence, UV-visible, pulsed amperometric) do not allow the determination of these three groups of compounds. However, this problem can be solved by means of the use of an ICP-AES Spectrometer [1].

The aim of the present work is to carry out the determination of carbohydrates, carboxylic acids and metals in a single chromatographic run by means of the coupling of High Performance Liquid Chromatography (HPLC) and ICP-AES in order to discriminate between several varieties of tomato according to their profiles.

2. Experimental conditions

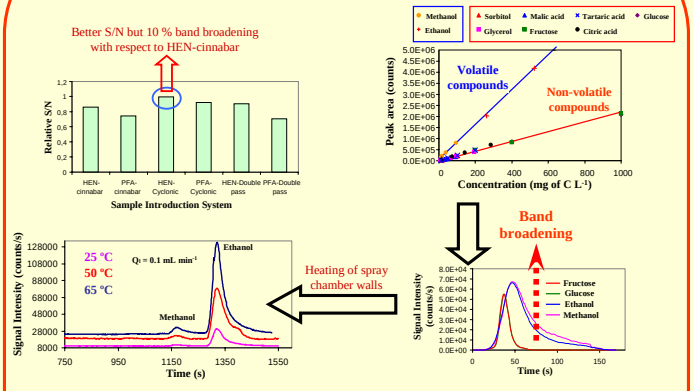
Spray chamber: Cinnabar® (Glass Expansion)
Outer gas flow rate: 15 L min^{-1}
Intermediate gas flow rate: 0.2 L min^{-1}
Central gas flow rate: 0.6 L min^{-1}
R.F. Power: 1.35 kW



Chromatographic conditions

Mobile phase flow rate, Q_l: 0.6 mL min^{-1}
Mobile phase: HNO_3 0.007 mol L^{-1}
Column: Rezex RHM-Monosaccharide H+ (8 %) (Phenomenex)
Column size: 300 x 7.8 mm
Column particle diameter: 8 mm
Guard cartridge: Carbo-H+ (Phenomenex)
Guard cartridge dimensions: 4 mm L x 3 mm ID
Oven temperature: 80 ºC

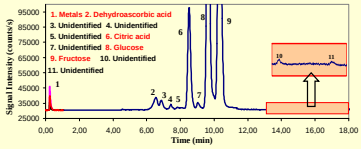
3. Study of detection conditions



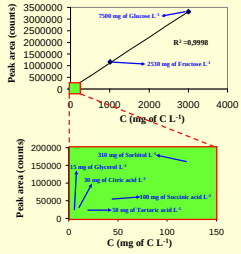
ICP-AES was characterized by its universality with regard to the detection of non-volatile compounds. Contrarily, volatile compounds such as methanol and ethanol showed higher sensitivities. However, a band broadening was observed for volatile compounds as a consequence of their evaporation from the spray chamber walls solution giving rise to worst chromatographic resolution. The heating of spray chamber walls did not reduce the peaks width because plasma was extinguished at heating temperatures below the boiling point of the compounds. Nonetheless an improvement in sensitivity was observed.

4. Analysis of tomato varieties

Chromatogram obtained for a tomato sample



SICA method

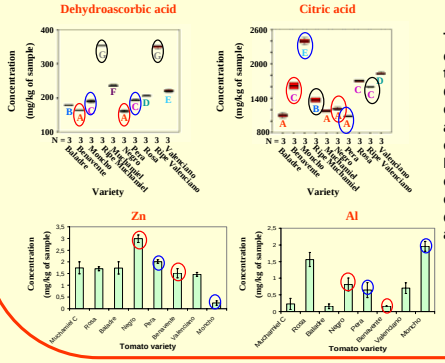


Comparison between SICA and conventional calibration

Multiple injection	Slope	Intercept
Tartaric acid	(217 ± 5)·10	(-2 ± 2)·10^3
Glucose	(208 ± 9)·10	(2 ± 5)·10^4
Citric acid	(230 ± 9)·10	(-3 ± 4)·10^3
Fructose	(21 ± 2)·10^2	(1 ± 9)·10^4
Glycerol	(20 ± 2)·10^2	(2 ± 6)·10^3
SICA	(213 ± 2)·10	(0 ± 3)·10^3

Universality of ICP-AES for the detection of organic compounds allows to carry out the calibration by means of the injection of only one standard containing increasing concentrations of a set of organic compounds representing the peak area against carbon concentration. The so-called Single Injection Calibration Approach (SICA) method permits to reduce considerably the time of analysis.

ANOVA ANALYSIS (BOX PLOT DIAGRAMS)



The profiles of carbohydrates, carboxylic acids and metals of tomato varieties permit their discrimination. Some varieties cannot be distinguished according to the concentration of a given organic compound. However, if other organic compounds or metals are considered their discrimination can be easily achieved.

5. Conclusions

-The coupling of HPLC to ICP-AES allows the simultaneous determination of organic compounds and metals thus providing information about food quality.
-Universality of ICP-AES with regard to the determination of non-volatile organic compounds allows to employ the SICA calibration method giving rise to a reduction in analysis time.
- The procedure developed has permitted the discrimination between different tomato varieties according to their profiles in carbohydrates, carboxylic acids and metals.

6. Acknowledgments

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[1] Paredes, E.; Maestre, S. E.; Prats, S.; Todoli, J. L.; *Anal. Chem.*; 2006; 78; 6774-6782.

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